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Crystallization of Polyetheretherketone (PEEK) in

Carbon Fiber Composites

by

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Carbon fiber has been found to act as a nucleating agent for crystallization of polyetheretherketone (PEEK). The number of ordered regions or nuclei in PEEK has been found to decrease as the total melt-annealing time is increased. Reducing the number of nuclei by long preheating in the melt favors PEEK crystallization on the carbon fiber, which makes a stronger interfacial bond and shows enhanced transverse tensile properties.

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CRYSTALLIZATION OF POLYETHERETHERKETONE (PEEK)

IN CARBON FIBER COMPOSITES

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## ABSTRACT

The tendency of carbon fiber to nucleate the crystallization of PEEK has been evaluated by DSC and other techniques. As the carbon fiber content was increased, the supercooling necessary for PEEK crystallization decreased. The repeated melting (at 396°C) of the same PEEK sample results in a decrease of the number of nuclei for crystallization. At equivalent thermal histories, PEEK with carbon fiber was found to have a higher nucleation density than PEEK itself.

The surface of carbon fiber and nuclei in the PEEK matrix compete for crystallization growth. As the holding time in melt was increased, the number of matrix spherulites formed on cooling decreased, hence a more pronounced transcrystalline region was developed. Correspondingly, the composites preheated in the melt for 100 min. showed about two times the transverse tensile strength and strain-to-failure of those preheated for only 30 min. Corresponding fracture surface produced in tension showed that the former samples had a greater matrix adhesion to the carbon fiber than the latter.

A strong interfacial bond is thus developed by crystallization on carbon fiber surface. Destroying nuclei in the PEEK matrix by long preheating enhances crystallization on the carbon fiber.

## INTRODUCTION

The fiber-matrix interface plays an important role in the mechanical properties of fiber-reinforced composites. Since the stress acting on the matrix is transmitted to the fiber across the interface, an evaluation of interface structure and properties is essential for an understanding of composite properties.

An early study of the interface in composites by Kardos et.al.[1,2] showed that the mechanical properties of a carbon fiber reinforced polycarbonate were improved by annealing. This was explained as due to the generation of a polycarbonate crystalline innerlayer at the fiber interface. Since the modulus of the crystalline structure is between that of the amorphous matrix and reinforcement, the crystalline interface may be a favorable medium for stress transfer. Another example of the improvement of mechanical strength using glass fibers coated with nucleating agents has been shown by Hobbs[3].

Nucleation on a carbon fiber surface has been reported for nylon 6[4], nylon 66[5], polyethylene[6], polypropylene[7] and polyetheretherketone[8]. However, studies of interfacial structure and its effects on mechanical properties have been limited.

Polyetheretherketone (PEEK) is a high performance thermoplastic resin of the chemical structure shown in Figure 1. Its glass transition temperature is ~143°C and melting point ~334°C and its crystallinity is <48%[9]. PEEK exhibits good resistance to attack by organic solvents[10].

The number of surviving nuclei in the melt has been found to depend on the temperature and time at which the polymer has been held in the melt temperature before cooling, which is a widely observed behavior of semicrystalline thermoplastics[11-14]. A cyclic DSC experiment, much as applied in this study, has been used to show that the repeated melting of the same sample of nylon 6 results in a decrease of nuclei for crystallization[15].

The longitudinal tensile strength (parallel to the fiber direction) of a composite is determined predominantly by the fiber strength. Conversely, the transverse tensile strength (perpendicular to the fiber direction) depends primarily on the interfacial strength between fiber and matrix[16]. Therefore, transverse tensile tests have been chosen for study of the adhesion between carbon fiber and PEEK.

## EXPERIMENTAL

The matrix polymer PEEK (Mn = 14,100, Mw = 38,600) was obtained from Imperial Chemical Industry (ICI). The carbon fiber, Thornel 300 (PAN-based, no finish) was obtained from Union Carbide. It was used without further treatment. PEEK powder was predried at 150°C in vacuo overnight before use. PEEK containing unidirectional carbon fibers was prepared in a hot press with a vacuum facility. Carbon fiber tows gripped at both ends were interleaved with previously-pressed amorphous PEEK films and compression molded at 390°C and 2 MPa, for 30 min. in vacuo. Before compression molding, two classes of samples (the SF and SS) were preheated for 30 min. and two other classes (the LF and LS) were preheated for 100 min. at 390°C without pressure. The compressed films were then cooled to room temperature in the press at fast (the SF and LF) or slow (the SS and LS) cooling (Table 1). The thickness of all composite films was ~0.22 mm.

The following DSC experiments were performed in nitrogen atmosphere using a Perkin-Elmer DSC-2 with a Thermal Analysis Data System. Approximately 10 mg samples in aluminum pans were used. The LS samples of various content of carbon fiber were heated to 396°C at 10°C/min. and immediately scanned on cooling. On cooling at -20°C/min., exothermic crystallization curves were recorded. The cyclic DSC experiment reported by Avramova et.al.[15] has been used to measure nucleation density. Samples were heated to 396°C at 10°C/min., and immediately cooled rapidly to 306°C and held there for 7 min. to follow PEEK crystallization. The samples were heated again to the same melt temperature and held for 20 min. for the 2nd cycle. The sample was again cooled rapidly to the same

crystallization temperature, where it was again held for 7 min. For each cycle, all variables were held constant except for the melt-annealing times, which were sequentially 0, 20, 20, 50 and 20 min. The melt annealing temperature was chosen in the range of the suggested PEEK processing temperature, 371 - 399°C[9].

To check that no crystallization occurred during the cooling to the crystallization temperature, the sample cooled to 306°C was immediately heat-scanned and no endotherm was observed. For each cycle, isothermal crystallization curves after the times indicated and melting peaks found on the heat-scan (10°C/min.) were recorded.

PEEK films (~20μ thick) containing several carbon fibers were pressed (2 MPa) between microscope cover glasses for 10 min. in a hot plate (390°C), and in vacuo followed by rapid cooling. The samples were held at 390°C in a small furnace in a nitrogen atmosphere and cooled at -0.5°C/min. On reaching 270°C the samples were quenched to room temperature to observe the morphology. Transcrystallinity developed on the carbon fiber surface, as well as spherulites in matrix, was observed using a Leitz optical microscope with cross-polarizers.

Rectangular strips (3 x 5 mm) of the four classes of samples (Table 1) were trimmed with a paper cutter and tensile tested using an Instron Universal Testing Machine. The tensile fracture surfaces were examined in an ETEC Autoscan Scanning Microscope (SEM) after coating with a ~200A thick gold layer in a Polaron E5100 SEM sputtering unit.

After the DSC experiments and tensile tests, the carbon fiber content of samples was measured by dissolving out the PEEK with concentrated sulfuric acid followed by neutralization, washing, and drying of the fibers.

## RESULTS AND DISCUSSION

The effect of carbon fiber on the crystallization of PEEK has been studied by DSC. The crystallization curves obtained during cooling are shown in Figure 2. As the carbon fiber content was increased, the crystallization of PEEK starts at ever lower supercooling suggesting that fibers act as a nucleating agent.

The isothermal crystallization curves at $306^{\circ}C$ for the neat PEEK-SF and for carbon fiber reinforced PEEK-SF are shown in Figures 3 and 4 respectively. The minima of the curves for both samples are shifted to longer time with increasing cycles. This procedure was described in the Experimental section. Both features suggest less bulk nucleation at longer melt-annealing times. The shift for neat PEEK is more sensitive to the prior thermal history than with carbon fiber. The dependency of PEEK crystallization on the holding time in the melt has been reported[17]. Three crystallization curves, each following 20 min. of melt-annealing, are shown in Figures 3 and 4. The minima of the curves are shifted to longer time with increasing cycles. This further indicates that crystallization behavior of PEEK depends not only on the previous melt-annealing time but shows a cumulative dependency on all prior melt-annealing times.

From the heat-scans following a 7 min. isothermal crystallization, the areas under the melting peak were measured. The peak areas were converted to % crystallinity using the heat of fusion given for fully crystalline PEEK, 31.1 cal/g[18], and plotted with total holding time in Figure 5. Significant differences in melting temperature for PEEK with and without carbon fiber has

not been observed. Without melt-annealing the samples reach about 25%
crystallinity. The crystallinity decreased with increased total melt holding
time. The crystallization of PEEK thus shows the cumulative effect of all prior
melt-annealing, as also reported for nylon 6[15]. After the final cycle, the
samples were found, on cooling (-20°C/min.), to have a crystallinity similar to
that of the virgin sample (31.8% and 33.4%, respectively). Similarly, it has
been found that crystallization of PEEK is not affected when exposed to nitrogen
at 375°C for 4 hrs[19]. This suggests that no appreciable crosslinking or degra-
dation occurred during melt-annealing time. It has been reported that PEEK is
thermally stable at 400°C for greater than 1 hr[9]. Thermal degradation has been
found by thermogravimetric analysis (TGA) to start at 550°C[20]. It is difficult
to measure the molecular weight because of the limited solubility of PEEK in
organic solvents at room temperature and the sulfonation reaction that occurs in
concentrated sulfuric acid.

There have been several explanations for effects of thermal history on
crystallization. Residual high molecular weight crystals or small crystals
enclosed in cracks or holes of foreign particles may survive temperatures higher
than the melting point[11,21]. Nuclei of subcritical size created by steady state
fluctuations in the melt may cause immediate crystallization on subsequent
cooling[22]. The exact origins of nucleation sites of polymers remain uncertain
for the present. Relatively ordered regions in a polymer melt may act as
nucleating sites[12,13]. Indeed, it has been found that PEEK retains some local
order at 380°C[23]. The thermodynamic melting point of infinite, perfect PEEK
crystals has been estimated to be 395°C[18].

Since the crystallization time (7 min.) is not sufficient for spherulites to impinge, the crystallinity developed in each cycle is considered to depend on the number of ordered regions, or nuclei, which have survived the previous melt-annealing. Therefore, the crystallinity is proportionally dependent on the nucleation density and the difference in nucleation density along the samples becomes more pronounced at long melt-annealing time. For the four classes of neat PEEK samples (Figure 5), the order of increasing nucleation density is found to be LS<SS<SF~LF. All PEEK samples with carbon fibers exhibit a higher nucleation density than PEEK itself at equivalent thermal histories. Carbon fiber reinforced LF sample shows a higher nucleation density than carbon fiber reinforced SF sample and the two samples without carbon fiber show a similar nucleation density, indicating that the contribution of carbon fiber to nucleation is greater in the LF sample than in the SF sample. The nucleation of polymer on substrates can be complex. Several different explanations have been offered including a consideration of surface energy of substrate[24], a possible temperature gradient[25], the matching of unit cell structure[26], and shear stresses[27].

Figure 6 shows cross-polar optical micrographs of PEEK crystallization in the presence of carbon fibers. For samples held in the melt for long times, fewer spherulites are seen in the bulk and a more distinctive transcrystalline region is developed on the carbon fiber surface. It is generally known that high density of nuclei at an interface promotes unidirectional growth of spherulites because of the proximity of nucleation sites. Noticeable transcrystalline structure did not develop in sample 1 (Figure 6), which had a thermal history

similar to the SS sample. The thermal history of sample 2 is similar to that of the LS sample. Here a ~5μ thick transcrystalline region impinged with the spherulites nucleated in the matrix. The crystallization on the carbon fiber competes with matrix crystallization. As the melt holding time was increased, the number of nuclei in the matrix decreased, favoring heterogeneous crystallization on carbon fiber. The nucleation density of PEEK has been found to decrease exponentially with increasing melt temperature[28]. Transcrystallinity was not observed in the sample which had the same thermal history as the LF and SF samples. Nonetheless, the heterogeneous crystallization on the carbon fiber in the LF sample is considered to be more favorable than in the SF sample, as shown in Figure 5. The thickness of transcrystalline region (~30μ) in sample 4 is almost the same as the radius of the largest spherulites in matrix, implying that carbon fiber surface and nuclei surviving in the bulk have almost the same activity.

To measure the interfacial bond, tensile tests were carried out in the transverse direction (perpendicular to fiber direction). The transverse tensile strength of a composite is usually less than the strength of matrix polymer. The low transverse strength of unidirectional laminates often limits the design of structures such as pipes for internal pressure. The transverse tensile strengths are plotted vs carbon fiber content in Figure 7. All composites show little dependence on fiber content over the test range.

The curve in Figure 7 represents the calculated transverse strength, assuming no interfacial bonding, i.e., that the fibers are regarded as cylindrical holes of a simple square array[16]. The SS and SF samples show values com-

parable to the calculated strength, indicating weak interfacial bonding.
However, the LS and LF samples show considerably higher values than the matrix,
indicating that they exhibit a strong interfacial bond between the carbon fiber
and PEEK. The corresponding average percent crystallinities are listed in Table
2. The transverse tensile properties are seen not to be sensitive to percent
crystallinity. The toughness, i.e., the area under the stress-strain curve, of
the LS and LF samples is ~5 times that of the SF and SS samples. This major
difference is considered to be due to crystallization on carbon fiber.
According to Keith and Padden[29], impurities which cannot crystallize diffuse
away from growing crystal surfaces. Therefore, if the matrix nucleation is
dominant, impurities will be accumulated at the fiber-matrix interface and the
interfacial bond will be weak. The interface, where the PEEK crystallizes pre-
dominantly by nucleation on the fiber, is thus expected to produce a strong
bond.

The effect of cooling rate was found to be minor. This is probably because
the transverse tensile properties of a composite depend on the crystalline
structure of the matrix as well as on the interfacial structure, since the fiber
content is low. During longer preheating, nuclei in the matrix of the LS and LF
samples are more extensively reduced than for the SS and SF samples. The inter-
face in the LS and LF samples is favorably crystallized by nucleation on the
fiber surface, forming a strong bond. Some of the SF and SS samples were sub-
sequently subjected to the same thermal history of the LF sample. They showed
tensile strength values similar to that of the LF sample, suggesting that the
thermal history above the melting point is the main reason for difference in

transverse tensile strength. The LS and LF samples show greater strain-to-failure (4.8, 4.4% respectively) than did the SF and SS samples (1.9, 1.7% respectively). Strain-to-failure of commercial ICI carbon fiber reinforced PEEK (50-55 vol%) has been reported to be 1%[30].

The fracture surface of the samples, resulting from transverse tensile testing are shown in Figure 8. In the SF and SS samples, carbon fibers are nearly bare showing poor wetting. In contrast, carbon fibers in the LF and LS samples show strong matrix adhesion, implying that failure was accompanied by matrix deformation, consistent with the corresponding enhanced mechanical properties.

## SUMMARY

Carbon fiber has been found to act as a nucleating agent for crystallization of PEEK. The number of ordered regions or nuclei in PEEK has been found to decrease as the total melt-annealing time is increased. Reducing the number of nuclei in the matrix favors PEEK crystallization on the carbon fiber, which makes a stronger interfacial bond as indicated by transverse tensile tests.

## ACKNOWLEDGEMENTS

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## TABLE 1

Compression Molding Condition of PEEK

and PEEK with Carbon Fiber

| Sample[a] Code | Preheating Time[b] (min.) | Cooling Rate ($^{o}$C/min.) |
|---|---|---|
| SF | 30 | -7 |
| SS | 30 | -0.6 |
| LF | 100 | -7 |
| LS | 100 | -0.6 |

[a] The first letter of sample code stands for preheating time:

    S (short) for 30 min and L (long) for 100 min

    The second letter stands for cooling rate:

    F (fast) at -7$^{o}$C/min and S (slow) at -0.6$^{o}$C/min.

[b] At 390$^{o}$C without pressure followed by compression molded

    at 390$^{o}$C and 2 MPa for 30 min.

## TABLE 2

Transverse Tensile Properties of PEEK Composites[a]

| Composite Code | Vol. % Fiber | Crystallinity[b] | Modulus | Strength | Strain-to -failure | Toughness[c] |
|---|---|---|---|---|---|---|
| | | % | GPa | MPa | % | $10^6 J/m^3$ |
| LS | 11-18 | 36 | 3.9 | 111 | 4.8 | 3.3 |
| LF | 17-27 | 42 | 4.3 | 106 | 4.4 | 3.3 |
| SF | 16-21 | 42 | 3.9 | 63 | 1.9 | 0.6 |
| SS | 16-32 | 45 | 4.0 | 60 | 1.7 | 0.4 |

[a]Strain rate was 0.033/min.

[b]By DSC using 31.1 cal/g for the heat of fusion for fully crystalline PEEK[18].

[c]Measured from the area under the stress-strain curve.

## FIGURE CAPTIONS

FIGURE 1:    Polyetheretherketone (PEEK)

FIGURE 2:    Crystallization curves on cooling (-20°C/min) from 396°C:

(a) 0.0; (b) 7.0; (c) 11.3; (d) 18.5 vol.% carbon in PEEK

FIGURE 3:    Isothermal crystallization of PEEK-SF. Melt-annealing times in

min at 396°C.

FIGURE 4:    Isothermal crystallization of 15.1 vol.% carbon fiber reinforced

PEEK-SF. Melt-annealing time in min at 396°C.

FIGURE 5:    Crystallinity at 306°C in 7 min vs. total melt-annealing time at

396°C: (□) PEEK-LF; (Δ) PEEK-SF; (0) PEEK-SS; (∇) PEEK-LS;

carbon fiber reinforced PEEK, (▬) LF 22.1 vol.%; (▲) SF 24.6

vol.%; (●) SS 23.9 vol.%; (▼) LS 20.7 vol.%

FIGURE 6:    Cross-polar optical micrographs of PEEK with carbon fibers:

samples held at 390°C in hr., (1) 0.5; (2) 2; (3) 3; (4) 4 hr and

cooled (-0.5°C/min) to 270°C, followed by quenching to room

temperature

FIGURE 7:    Transverse tensile strength vs. carbon fiber content: (●) LS;

(Δ) LF; (□) SF; (▼) SS. The predicted transverse tensile

strength is $\sigma_m[1-2 \ (V_f/\pi)^{1/2}]$ where $\sigma_m$ is the tensile strength of

the matrix and $V_f$ is the vol. content of fiber[16]. 100 MPa[9] was

obtained for the PEEK-SF film and used for $\sigma_m$

FIGURE 8:    Scanning electron micrographs of tensile fracture surfaces of the

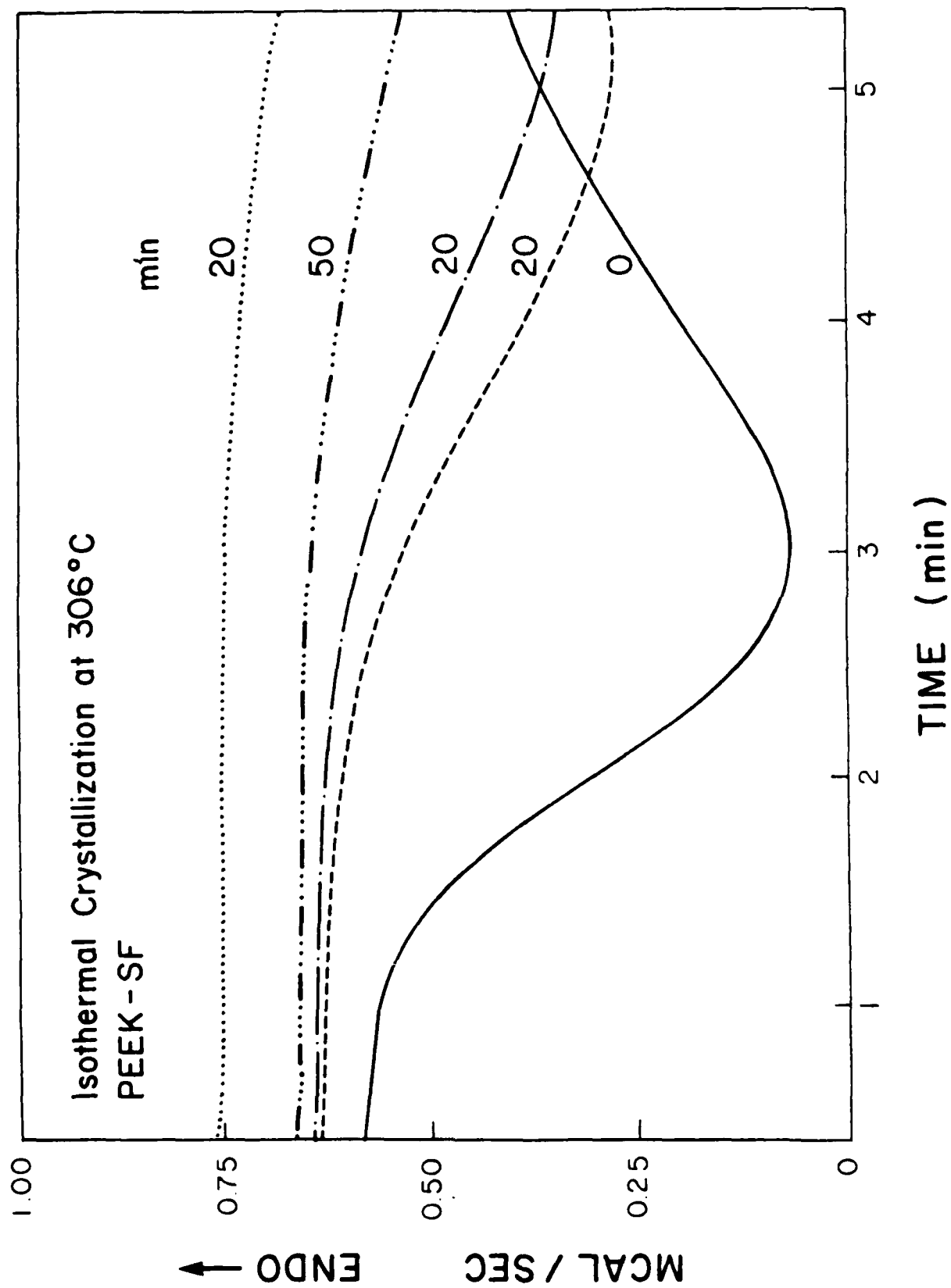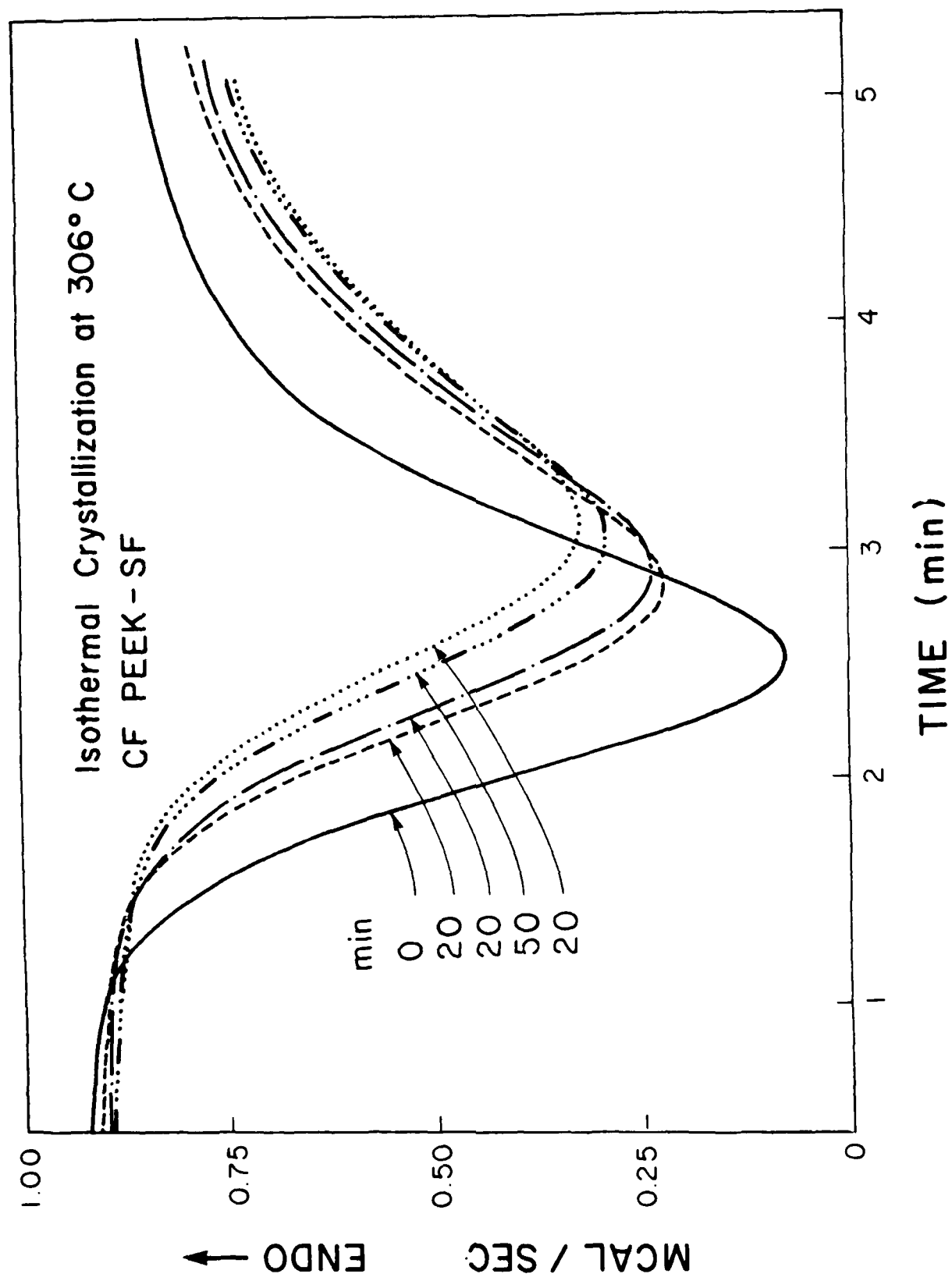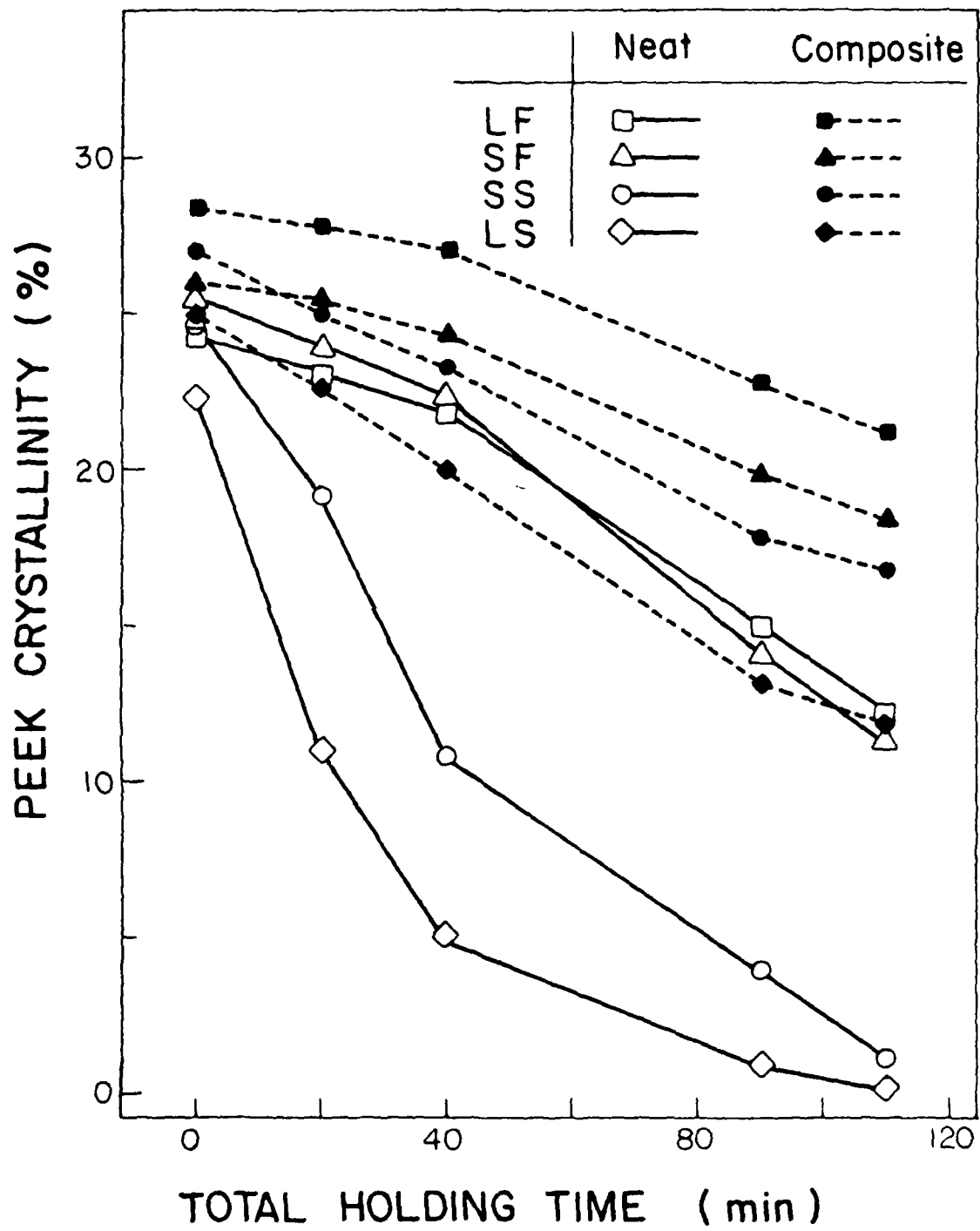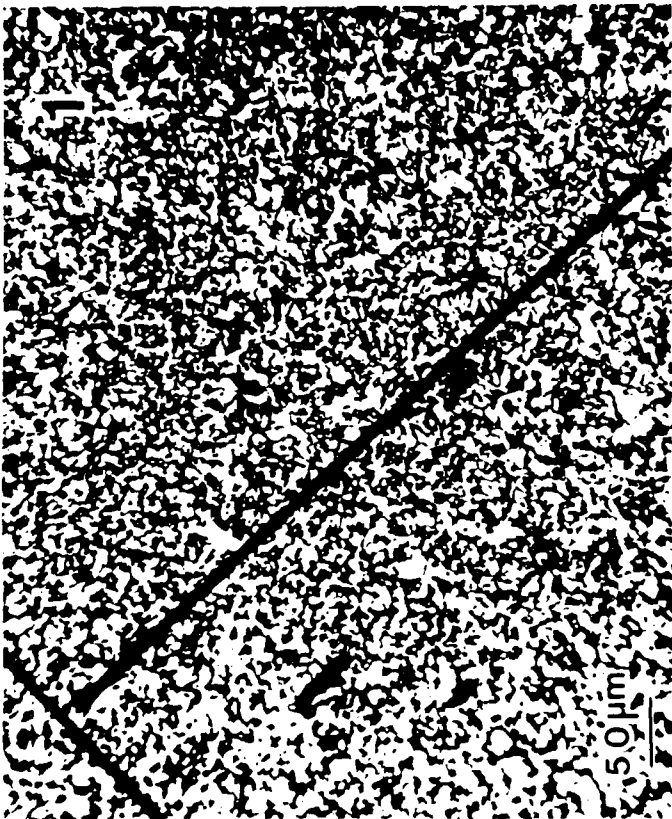SF, SS, LF, and LS sample

# Poly ether ether ketone （PEEK）

CF vol %

| | |
|---|---|
| a | 0.0 |
| b | 7.0 |
| c | 11.3 |
| d | 18.5 |

ENDO ←

MCAL/SEC

TEMPERATURE (°C)

265  270  275  280  285  290  295

Figure 3

Isothermal Crystallization at 306°C
PEEK-SF

MCAL/SEC    ENDO →

Figure 4.

Isothermal Crystallization at 306° C
CF PEEK-SF

min
0
20
20
50
20

TIME (min)

MCAL / SEC.

← ENDO

Figure 5

1

2

3

4

50μm
50μm
50μm
50μm
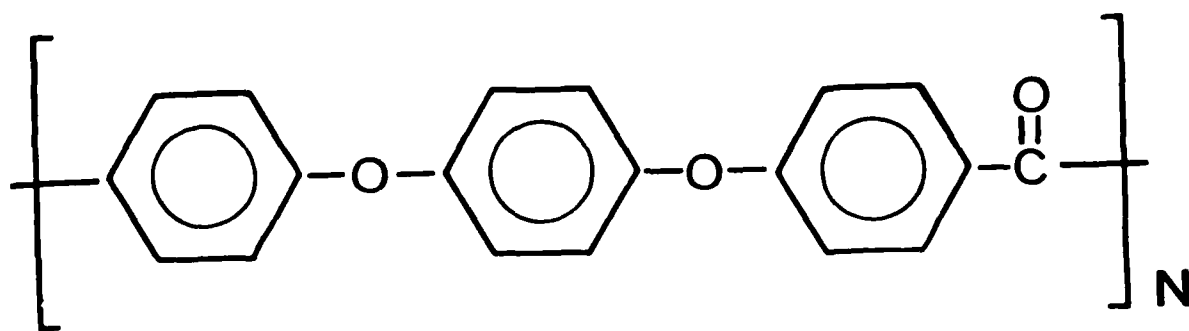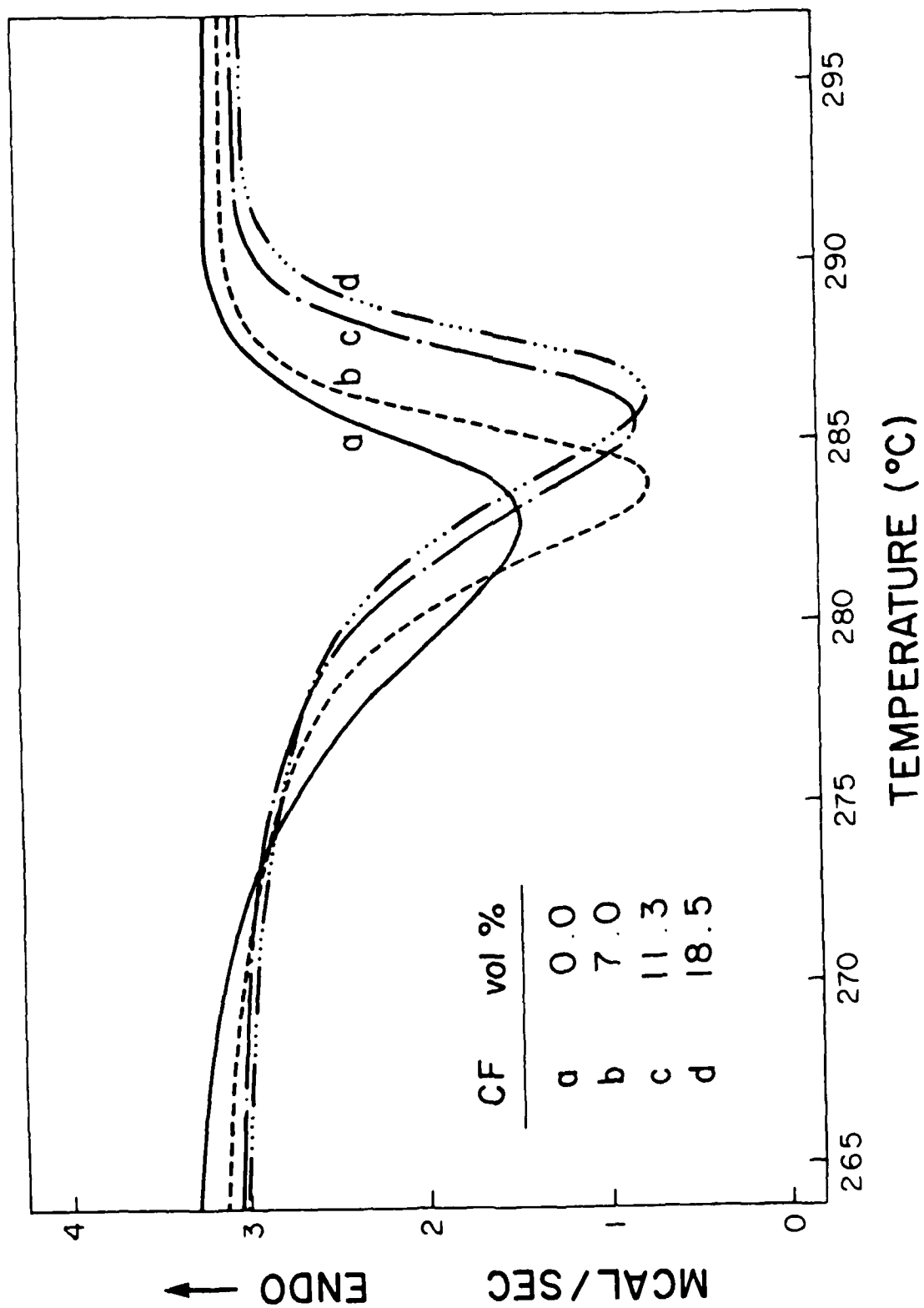
TRANSVERSE TENSILE STRENGTH (MPa)

Calculated

LS
LF
SF
SS

# END

# FILMED

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